C(15)	0.9482 (3)	0.2275 (11)	0.8944 (6)	0.105 (3)
C(16)	0.8899 (3)	0.1219 (8)	0.8330 (4)	0.082 (2)
C(17)	0.7988 (3)	−0.0877 (7)	0.5103 (4)	0.083 (2)

Table 2. *Selected geometric parameters* (Å, °)

Fe—C(1)	2.007 (4)	Fe—C(8)	2.077 (4)
Fe—C(2)	2.026 (4)	Fe—C(9)	2.071 (4)
Fe—C(3)	2.083 (4)	Fe—C(10)	2.017 (3)
Fe—C(4)	2.080 (4)	Si—C(1)	1.879 (4)
Fe—C(5)	2.019 (4)	Si—C(6)	1.880 (4)
Fe—C(6)	2.003 (4)	Si—C(11)	1.858 (4)
Fe—C(7)	2.028 (4)	Si—C(17)	1.856 (5)
C(1)—Si—C(6)	96.2 (2)	C(11)—Si—C(17)	112.4 (2)
C(1)—Si—C(11)	109.3 (2)	Si—C(1)—C(2)	120.4 (3)
C(6)—Si—C(11)	111.5 (2)	Si—C(1)—C(5)	117.7 (2)
C(1)—Si—C(17)	113.3 (2)	Si—C(6)—C(7)	118.3 (3)
C(6)—Si—C(17)	113.2 (2)	Si—C(6)—C(10)	119.6 (3)

H atoms were placed in calculated positions (C—H 0.96 Å) and included in the refinement as riding atoms with a general isotropic displacement parameter which refined to 0.093 (4) $Å^2$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *XCAD-4 Software* (Siemens, 1993). Program(s) used to solve structure: *SHELXTL/PC XS* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXTL/PC XLS*. Molecular graphics: *SHELXTL/PC XP*. Software used to prepare material for publication: *SHELXL93* (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1039). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Stoeckli-Evans, H., Osborne, A. G. & Whiteley, R. H. (1976). *Helv. Chim. Acta*, **59**, 2402–2406. [Note: unit-cell parameter *a* reported as 14.18 (2) should be 14.81 (2) Å.]

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The Twinned Crystal Structure of Bis(η^6-mesitylene)ruthenium(II) Tetrafluoroborate at 150 K

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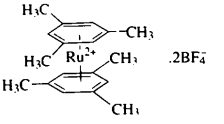
Abstract

The title compound, [Ru(C_9H_{12})$_2$]2BF_4, forms cubic crystals twinned on (110). The [Ru(mesitylene)$_2$]$^{2+}$ cation resides on a $\bar{3}$ special position and therefore adopts a staggered conformation.

Comment

There is considerable interest in the structures and properties of bis(η^6-arene) complexes of the transition metals. Both the physical and chemical properties of these materials may be readily modified and this has led to their use as components in one-dimensional charge-transfer conductors. Bis(arene) complexes typically exhibit very low energy barriers to changes in the relative orientation of the arene ligands. The conformation that these complexes adopt is therefore subject to crystal packing forces and this furnishes a potential tool for the fine tuning of their properties. Bis(benzene)ruthenium(II) dications have been employed in several one-dimensional solids (Suravajjala, Polam & Porter, 1993) and we now report the crystal structure of its close relative, bis(η^6-mesitylene)ruthenium(II) tetrafluoroborate, (1) (mesitylene is 1,3,5-trimethylbenzene), derived from a crystal twinned on (110).



(1)

In the solid state, compound (1) consists of discrete [Ru(mesitylene)$_2$]$^{2+}$ cations (Fig. 1) and BF_4^- anions, with bond distances and angles as listed in Table 2. In common with many 1:2 salts, compound (1) adopts the fluorite structure (Fig. 2) with the site symmetries of the cation and anion reduced from $m\bar{3}m$ and $\bar{4}3m$ to $\bar{3}$ and 3, respectively. The cations thus adopt the same staggered conformation as in [Ru(mesitylene)$_2$]$^{2+}$[$C_6(CN)_6$]$^{2-}$, (2)

(Ward, 1987). The bonds from Ru to the methylated C atoms are slightly longer [2.251 (5) Å] than those to the unsubstituted C atoms [2.209 (5) Å] and there is no significant bond-length alternation within the rings. The deviations of atoms C(1), C(2) and C(3) from the mean plane of the C_6 ring are −0.016 (4), 0.016 (4) and 0.006 (7) Å, respectively. These features are also observed in compound (2) and the geometric parameters associated with the cations do not differ significantly in these two salts. The BF_4^- anions exhibit typical bond distances and angles.

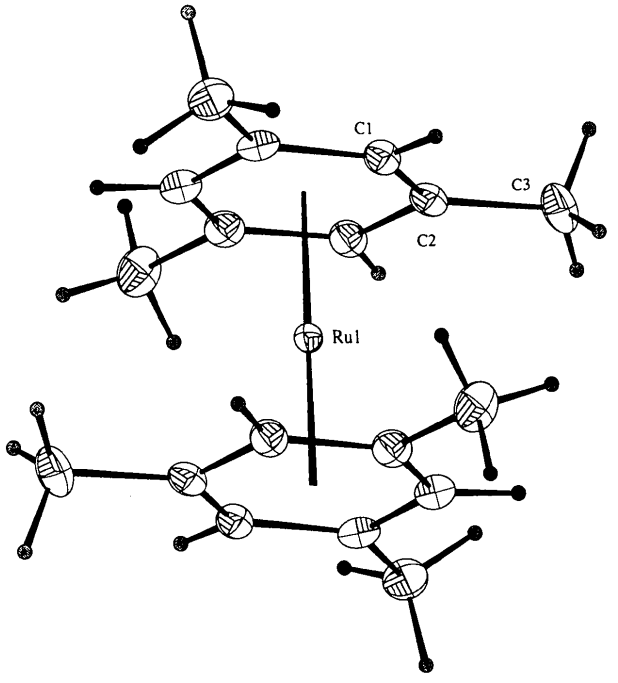


Fig. 1. A view of the $[Ru(mesitylene)_2]^{2+}$ cation with the atom-numbering scheme. Displacement ellipsoids enclose 50% probability surfaces.

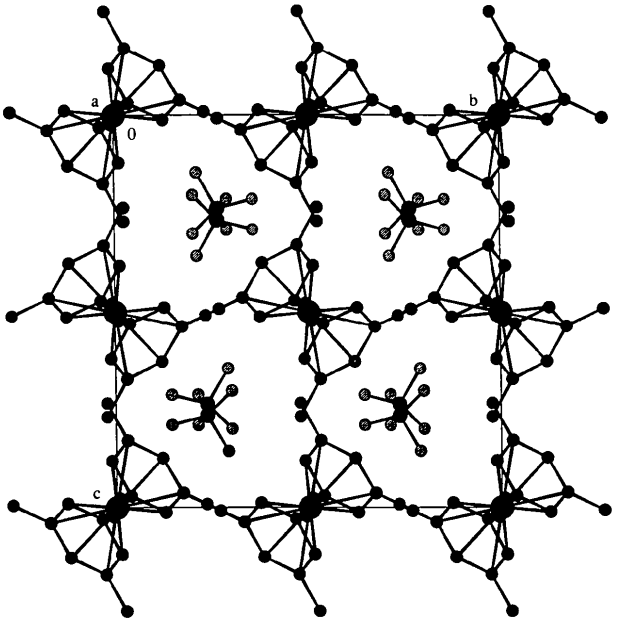


Fig. 2. A packing diagram of (1) showing the adoption of the fluorite structure.

Experimental

The title compound was prepared according to the literature method (Fischer & Boltcher, 1957) and well formed colourless octahedral crystals were grown by slow evaporation from nitromethane at room temperature.

Crystal data

$[Ru(C_9H_{12})_2]2BF_4$
M_r = 515.06
Cubic
$Pa\bar{3}$
a = 12.636 (3) Å
V = 2017.6 (8) Å^3
Z = 4
D_x = 1.696 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 42 reflections
θ = 15.5–16°
μ = 0.850 mm^{-1}
T = 150.0 (2) K
Octahedron
0.74 × 0.66 × 0.66 mm
Colourless

Data collection

Stoe Stadi-4 four-circle diffractometer
ω–2θ scans with ω-half-width (1.5 + 0.35tanθ)°
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
T_{min} = 0.306, T_{max} = 0.574
2050 measured reflections
639 independent reflections
491 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.128
θ_{max} = 25.01°
h = 0 → 15
k = 0 → 15
l = 0 → 15
3 standard reflections
frequency: 60 min
intensity decay: 1.9%

Refinement

Refinement on F^2
$R(F)$ = 0.0412
$wR(F^2)$ = 0.1820
S = 1.171
599 reflections
46 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 3.2944P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.025
$\Delta\rho_{max}$ = 0.582 e Å^{-3}
$\Delta\rho_{min}$ = −1.227 e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Ru1	1/2	1/2	1/2	0.0169 (4)
C1	0.4878 (4)	0.6198 (4)	0.6268 (4)	0.0284 (11)
C2	0.5376 (4)	0.5295 (5)	0.6716 (4)	0.0296 (11)
C3	0.4915 (4)	0.4767 (5)	0.7675 (5)	0.0401 (15)
F1	0.2887 (4)	0.2108 (3)	0.3554 (3)	0.0725 (13)
F2	0.2010 (2)	0.2010 (2)	0.2010 (2)	0.0382 (13)
B	0.2645 (5)	0.2645 (5)	0.2645 (5)	0.030 (2)

Table 2. *Selected geometric parameters* (Å, °)

Ru1—C1	2.209 (5)	C2—C3	1.500 (8)
Ru1—C2	2.251 (5)	F1—B	1.368 (5)
C1—C2^{i}	1.407 (8)	F2—B	1.390 (12)
C1—C2	1.420 (9)		
C2^{i}—C1—C2	121.9 (5)	C1—C2—C3	120.5 (5)
C1^{ii}—C2—C1	118.0 (5)	F1^{ii}—B—F1	109.9 (4)
C1^{ii}—C2—C3	121.4 (6)	F1—B—F2	109.1 (5)

Symmetry codes: (i) y, z, x; (ii) z, x, y.

Preliminary microscopic examination of the crystals revealed that they did not transmit polarized light in any orientation, implying that they were cubic. This was confirmed on indexing a list of reflections generated by a random search of reciprocal space made on a Stoe Stadi-4 four-circle diffractometer. Data were collected at 150 K using an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986).

Examination of the full data set, which had been corrected for Lp and absorption effects, implied a primitive unit cell in Laue group $m\bar{3}m$ with reflections in the classes $hk0$, $h0l$ and $0kl$ systematically absent only when both indices are odd. Reflections with all indices even or all indices odd tended, in addition, to be systematically strong, a symptom of pseudo-F centring. These observations suggested that the crystal was twinned on (110) in space group $Pa\bar{3}$, with the Ru atoms occupying positions equivalent to 0,0,0 or $\frac{1}{2},\frac{1}{2},\frac{1}{2}$, and common for both twin components. Thus, whereas reflections of the class $hk0$ are normally present for $h = 2n$ in this space group, inclusion of the twin component, which interchanges h and k, means that this absence condition only holds when both indices are odd.

The volume of the unit cell (2017.6 Å^3) implied the presence of four formula units of (1) per unit cell. Interpretation of the Patterson function was consistent with an Ru atom at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ and suggested a position for a C atom to break the pseudo-symmetry. Subsequent Fourier syntheses with coefficients $(2F_o-F_c)$ and (F_o-F_c) located the BF_4^- and remaining C-atom positions, respectively. During the initial stages of least-squares refinement the geometries of the mesitylene and tetrafluoroborate fragments were made subject to chemically reasonable restraints (bond lengths: C—C = 1.39 and 1.52, B—F = 1.4 Å) and the twin component was allowed to vary. The restraints were later removed and the twin component fixed at 0.5 after it had refined to 0.498 (5).

The H atom attached to the ring was placed in a calculated position (C—H = 0.95 Å), and allowed to ride on C(1). The positions of the methyl H atoms were derived from a difference synthesis. During refinement the C(3)—H bond lengths (0.98 Å) and H—C(3)—H angles (109.5°) were fixed but the H—C(3)—C(2)—C(1) torsion angles were allowed to vary. All H atoms were assigned an isotropic displacement parameter equal to $1.2U_{eq}$(C).

Data collection: *DIF*4 (Stoe and Cie, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1990*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *CAMERON* (Pearce & Watkin, 1993).

We thank the SERC for provision of a four-circle diffractometer and the Univ. of Edinburgh for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: MU1142). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Tungsten Nitrosyl Complex

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Abstract

Insertion of nitric oxide into one W—C bond of a bis(trimethylsilyl)tungsten complex results in the formation of a bidentate *N*-trimethylsilylmethyl-*N*-nitrosohydroxylaminato ligand. The resulting complex, (η^5-cyclopentadienyl)nitrosyl(trimethylsilylmethyl)(*N*-trimethylsilylmethyl-*N*-nitrosohydroxylaminato)tungsten, $[W(NO)(C_5H_5)(C_4H_{11}N_2O_2Si)(C_4H_{11}Si)]$, crystallizes in space group $P\bar{1}$, Z = 4 (two molecules per asymmetric unit). The two independent molecules are almost identical and have a four-legged piano stool structure with a five-coordinate W atom.

Comment

The ligand geometry is similar to that found in $[WMe_4\{ON(Me)NO_2\}]$ (Fletcher, Shortland, Skapski & Wilkinson, 1972; Fletcher & Skapski, 1973).